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Determination of the alpha-energy of U^{235}

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With 9 figures in the text

I. Introduction

Various methods have been used to determine the isotope abundance ratios of enriched samples of uranium. (1). One of them, using alpha-ray analysis, also allows the determination of the alpha-energy of U^{235}; this method is described below.

Since the three isotopes under consideration, U^{234}, U^{235} and U^{238}, are natural alpha-emitters, comparison of abundance ratios can be made by means of an electronic alpha-ray spectrometer, consisting of an ionization chamber and a linear amplifier connected to a multi-channel pulse amplitude analyser ("Kick-sorter"). The pulses due to the alpha-particles are sorted into three groups, the number in each group being a measure of the relative abundance of the respective isotope, and the position a measure of the energy. To fix the energy scale of the spectrometer one needs at least two wellknown alpha-groups, preferably lying close to the unknown one, and for this purpose U^{234} and U^{238} may be used.

The whole difficulty in such a measurement consists in obtaining sufficient resolution and stability of the rather complicated apparatus to ensure that the very scarce impulses from U^{235} (approximately 1.8 % of the total number) are not hidden entirely in the tails of the large peaks due to U^{234} and U^{238}. Since this difficulty has been overcome by means of certain details in the design of the apparatus, a brief description of this follows.

II. Apparatus

The entire experimental equipment is shown schematically in Fig. 1.

The ionization chamber (Fig. 2) is of the grid type (2) (3), and is worked with electron collection in pure argon at a pressure of approximately 1 atmosphere. The supply voltage, of the order 1 kV, is carefully filtered by means of an $R-C$ network, and divided in the right proportion for the grid. The high voltage electrode may also be connected to the pulse generator in order to check the working of the electronic part of the equipment. The uranium samples are electrolytically deposited, probably in the form of UF_4 (4), on circular discs of stainless steel, finished to semi-optical polish. The uranium

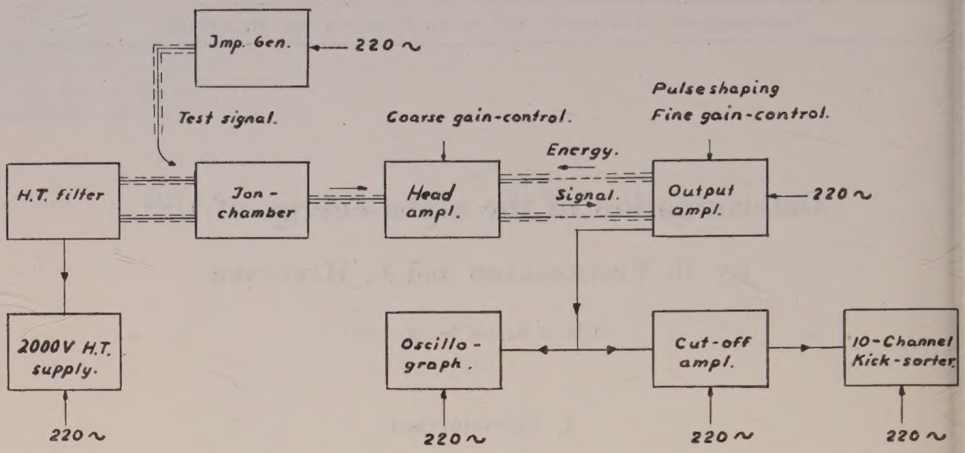


Fig. 1. Block diagram of the experimental equipment.

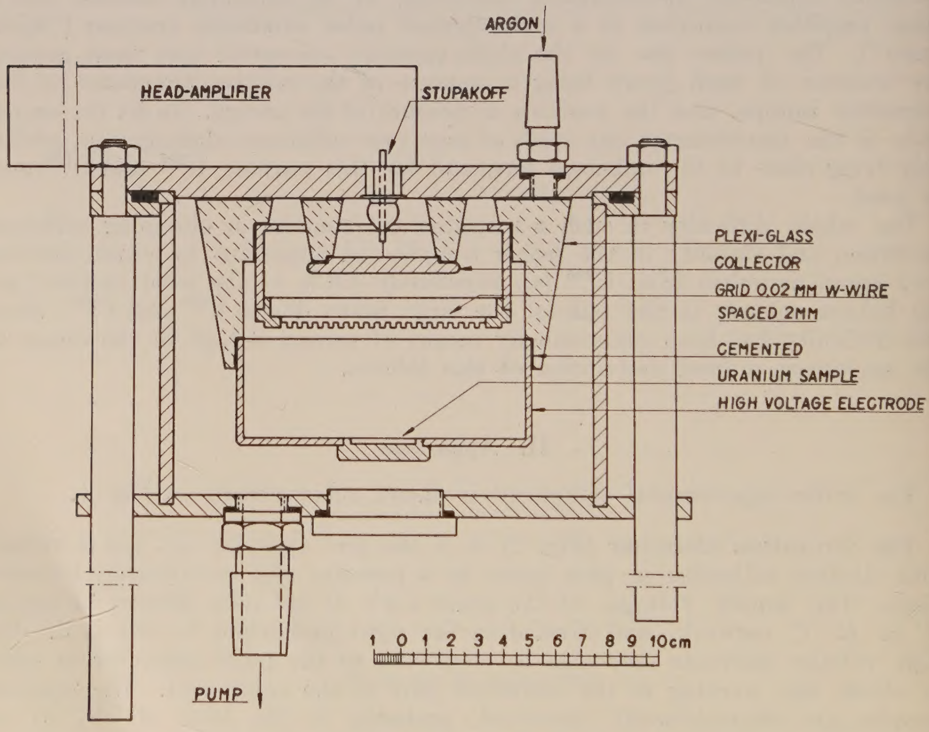


Fig. 2. The ionization chamber

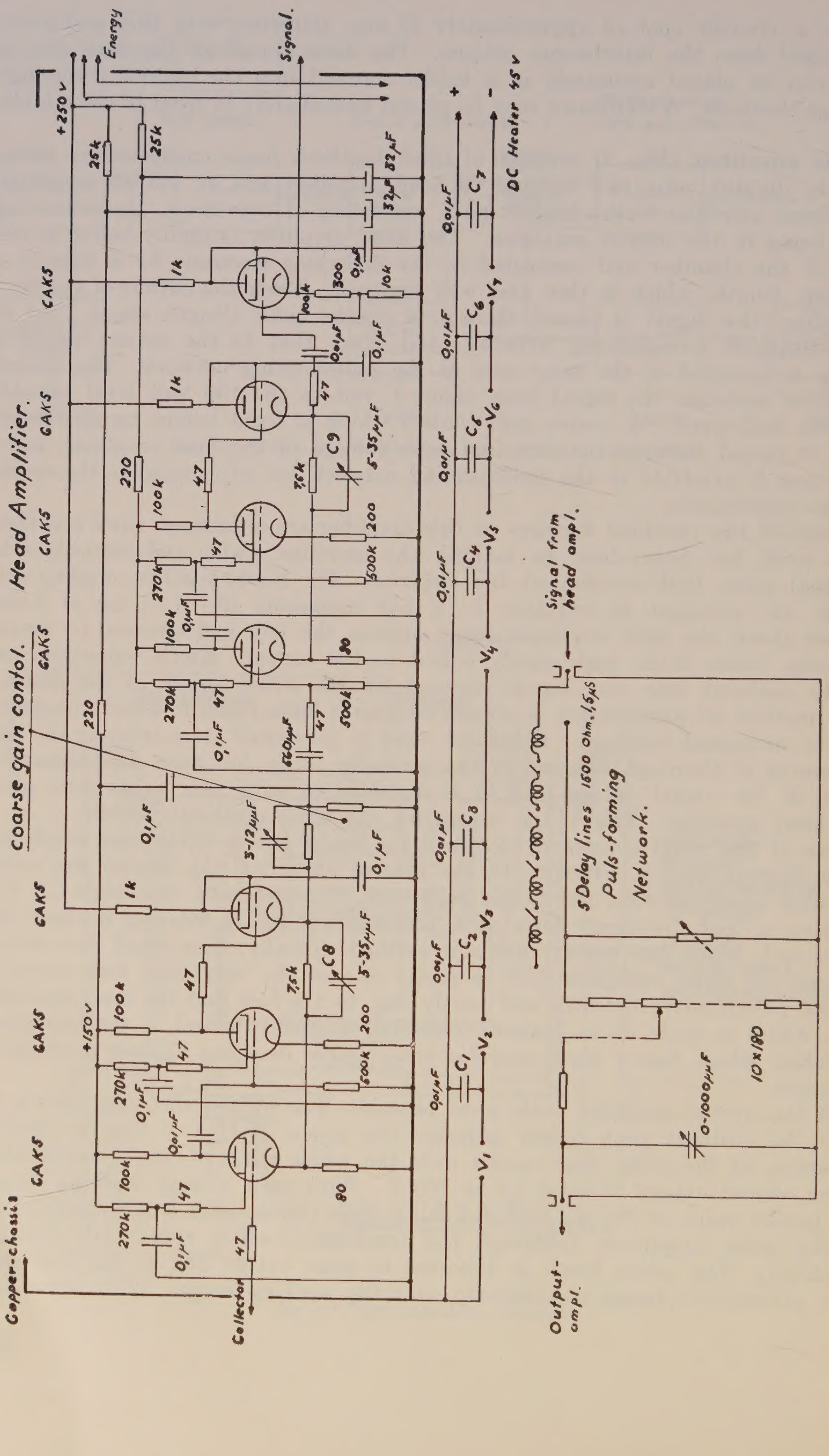
forms a circular spot of approximately 25 mm diameter, very thin and even, as judged from the interference colours. The discs are all of the same size, so they can be placed accurately in a holder screwed into the bottom of the high voltage electrode. A collimator may be placed immediately in front of the sample.

The amplifier (Fig. 3) consists of three feedback loops connected in series, and is divided into two sections, a head amplifier and an output amplifier. The head amplifier is the biggest part, consisting of two loops, the remaining loop being in the output amplifier. The head amplifier is rigidly bolted to the top of the chamber and connected to the collecting electrode by a wire of a few cm. length, which is thin and well screened. From the output of the head amplifier, the signal is passed through a coaxial cable (length about 2 m) to an adjustable pulseforming network, and from that to the output amplifier which is mounted in the same case as the pulseforming network. The output amplifier increases the signal from about 1 volt to the 100 volt level suitable for the kick-sorter. A coarse gain control which is fixed before measurements start is placed between the first and second loops of the head amplifier, and a finer one is available in the pulseforming network for adjustment in the course of the experiments.

Some of the practical features of this amplifier are of interest, since considerable work has been done in making the amplifier stable and insensitive to external noise, both mechanical and electrical. The head amplifier coupling networks are arranged in sequence in a box measuring 45 × 8 × 5 cm of 2 mm copper sheet, the tube envelopes being outside the box and screened by means of brass tubes 5 cm high rigidly bolted to the copper. Earth connectors are firmly soldered into the copper throughout the entire length of the chassis. This method of construction is chosen because it helps much to avoid instability due to unwanted feedback. Electrical noise is prevented from entering the box by means of thorough filtering in the incoming leads, but since this cannot be done in the signal output lead, it is desirable to have only a low gain after the head amplifier. This is the reason for the unconventional layout.

One of the worst sources of trouble is the heater circuit of the head amplifier, since filtering here is difficult. In the present amplifier, the heaters are series fed with direct current, the heater resistance, together with capacitors C_1—C_7, forming a high frequency filter with progressively better filtering towards the input end, where the heater circuit is earthed. Finally, it is found that microphonics are quite negligible: this is partly due to the very short time constant in the pulseforming network and partly due to the fact that the head amplifier box, which is made of an inelastic material, is rigidly bolted to the ionization chamber, whose heavy mass provides considerable damping against mechanical vibration.

Of the output amplifier little need be said. The biggest difficulty here is to have the feedback path rightly adjusted (by means of C_{10}) so that no serious distortion of the pulse front occurs over the whole range of output amplitudes, which should extend at least up to 100 V. With such a large working range, the proper value of C_{10} for highest-fidelity pulse transmission is not independent of the pulse amplitude (although the amplitude itself is very nearly linearly amplified). The pulse front is distorted to some extent due to the fact that tube parameters change considerably over the working range. With some care


Head Amplifier.
Coarse gain contol.
fine gain control.
Copper-chassis
Collector
Energy
Signal.
+250 v
+150 v
6AK5
6AK5
6AK5
6AK5
6AK5
6AK5
6AK5
25k
25k
1k
1k
1k
220
220
100k
100k
100k
100k
270k
270k
270k
270k
47
47
47
47
47
47
47
47
0,1 μF
0,01 μF
32 μF
32 μF
300
10k
100k
200
200
500k
500k
500k
80
80
7,5k
7,5k
C9
C8
5-35 μμF
5-35 μμF
3-12 μμF
560 μμF
0,01 μF
C1
C2
C3
C4
C5
C6
C7
C8
V1
V2
V3
V4
V5
V6
V7
+
-
DC Heater 45 v
Signal from head ampl.
5 Delay lines 1600 Ohm, 1,5 μS
Puls-forming Network.
0-1000 μμF
10×180
Output-ampl.

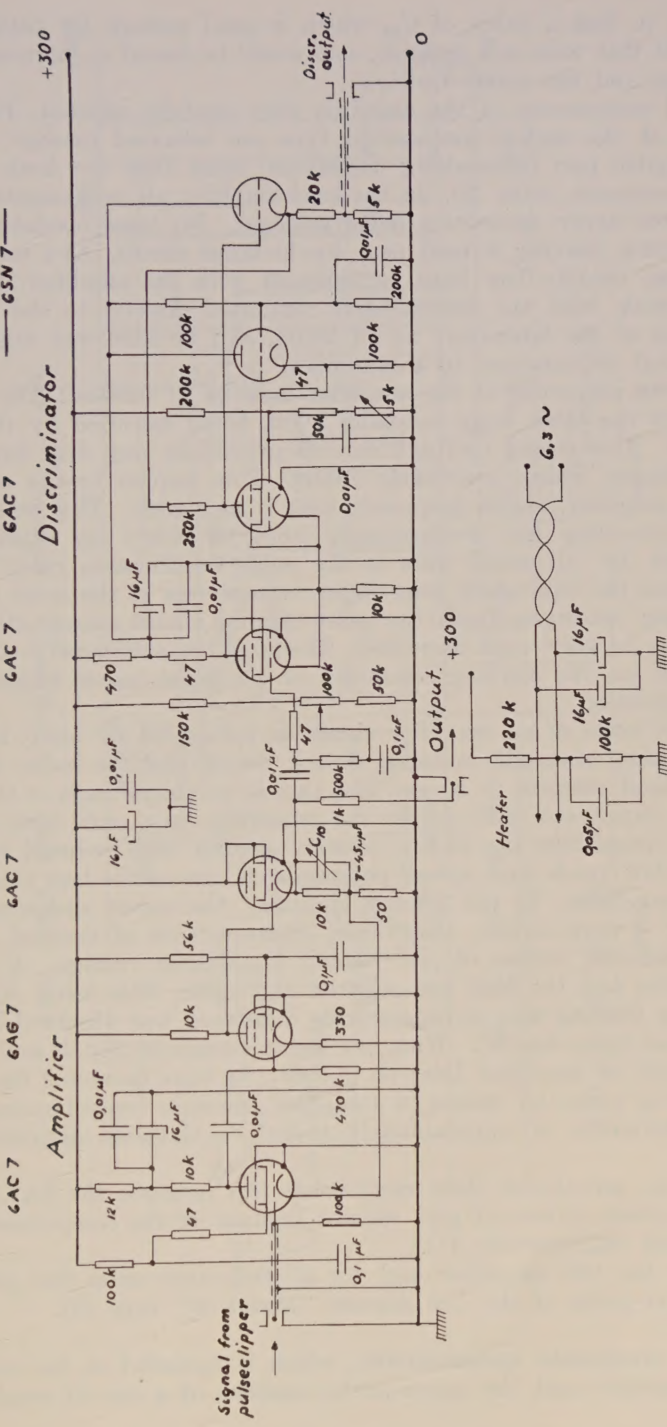


Fig. 3 b. Output amplifier and pulse discriminator.

own design (Fig. 4) (9) and a ten channel "kick-sorter" (Fig. 5). The cut-off amplifier is intended to cut off the lower part of the pulses, and amplify only their tops, so that the amplitude region of interest is spread across the whole range of the "kick-sorter". Quantitatively, the operation of this unit is described to the first approximation by

$$A_0 = (A_i - V_0) \cdot G,$$

A_0 and A_i being respectively output and input pulse-amplitudes, V_0 a threshold voltage (0–75 V) and G a magnification (values 1, 2, 3, 4 and 5). At the same time one obtains a certain amount of "smearing", only the leading edge of the pulse between V_0 and the pulse maximum being reproduced in the output voltage. The decaying part is never faster than an exponential with time constant of 10 μs.

The "kick-sorter" is an old design, which is limited in counting rate by the mechanical registers, and its ten channels are far too few for getting a detailed spectrum of the three groups under discussion. As a consequence of this we have been forced to construct the spectra out of repeated overlapping measurements. The whole procedure is extremely time-consuming and demands an apparatus of very high stability.

The pulse generator (Fig. 6) delivers a pulse of very constant amplitude at a certain rate not too fast for the registers, and is used for regular tests of the whole electronic equipment. The pulse, which is negative-going, has an amplitude of about 1 V. Its front has a rise time of $4 \cdot 10^{-8}$ sec. and the top is absolutely flat for 400 μs followed by a decay of 50 μs time constant. The amplitude can be read on an accurate meter (M in Fig. 6).

This test pulse is applied to the high-voltage electrode of the chamber and induces a charge upon the collector through the extremely small capacitance existing between these electrodes. The capacitance is so small ($\frac{1}{500}$ of the total input capacitance) that

$$q = C_T \cdot V,$$

q being the charge induced on the collector by a pulse of amplitude V applied to the test capacitance C_T, which is the natural capacitance between high-voltage electrode and collecting electrode, with the grid grounded.

This pulse test provides an ideal check upon the electronic apparatus under exactly the same working conditions as exist during the measurements. It should reveal any fault or change in the entire apparatus, short of changes in the ion-collecting process in the chamber. The output of the impulse generator has been adjusted so as to induce a charge on the collector of about the same magnitude as those caused by the alphas and the test has been applied before and after very single measurement throughout the entire working period.

Before measurements started the pulse test system was used to determine the amplifier constants, the charge sensitivity of the chamber-plus-amplifier system, the amplifier noise amplitude spectrum, and the efficiency of the chamber which is here defined as the number of electrons collected per alpha track

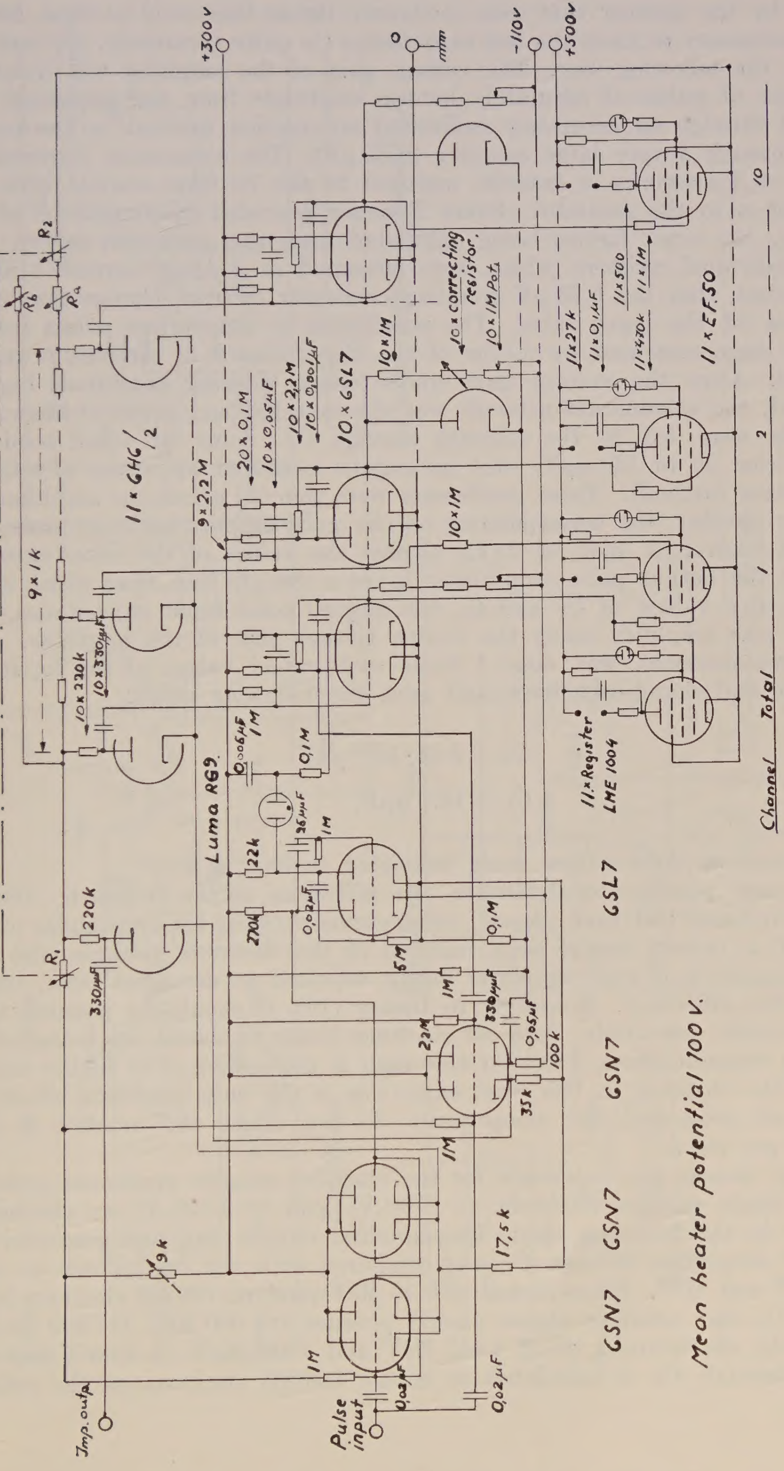


Fig. 5. 10-channel "kick-sorter".

divided by the number that were produced. Before this could be done, however, it was necessary to know the test capacitance C_T quite accurately. C_T was measured in the following way: The voltage gain of the amplifier was determined by means of pulses of accurately known amplitude from the generator. They were fed through an accurately calibrated attenuation network to the amplifier input, through a very large capacity (0.25 μF). The attenuator consisted of a number of T-networks in cascade, matched to the 70 Ohm coaxial cable which connected it to the generator. Every T-section provided an attenuation of about 10 times, the exact figures being calculated from the accurately-known values of the individual resistors, which were measured in a direct current bridge. It was checked, that the 0.25 μF coupling condenser did not change the working conditions of the input valve. The amplitudes of the output pulses from the amplifier were measured by means of the discriminator in the output amplifier (Fig. 3 b). After the voltage gain under exact working conditions had been calculated, the attenuation network was disconnected and pulses of known fixed amplitude were fed to the collector through C_T. Some ten small condensers, ranging from 20 to 120 μμF, were accurately measured by means of a Q-meter (substitution method). These condensers were shunted across the amplifier input one after another, the corresponding output pulse amplitudes being measured as described above. A plot of $1/V_0$ against the values of the shunt capacitors, V_0 being the output pulse amplitudes, gives a straight line, from which one can calculate the values of C_T and C_i, the natural total input capacitance of the chamber plus amplifier, using the known voltage gain of the amplifier.

This measurement was done 4 times with varied values of the input pulse amplitude and shunt capacitors, and gave the following result:

$$C_T = 2.35 \cdot 10^{-2}\ \mu\mu\mathrm{F}$$

$$C_i = 15.7\ \mu\mu\mathrm{F},$$

the accuracy of these values being estimated to be ± 5 %.

It is now possible to determine the efficiency of the chamber. After the working voltages had been chosen, using curves showing the amplitudes of alpha pulses of a certain energy as a function of the electrode voltages, the pulse-forming network of the amplifier being adjusted as described later, one can measure the efficiency. According to theory (10), it should be possible to find such electrode potentials, that all electrons being produced are collected (disregarding recombination, which in this case is negligible); it is highly desirable to work the chamber in this way, since this is the only condition where small changes of potentials and temperature do not effect the amount of charge collected per track.

Having chosen the potentials for the chamber to give maximum pulse amplitudes (high voltage electrode — 1 700 V, grid — 1 100 V) we checked the efficiency in the following way: The amplifier output due to a generator pulse of 1.00 V amplitude through C_T was compared with the output due to alphas from U^{234} and U^{238}. Since alphas of 5.30 MeV produce 186 200 electrons in pure argon (11), the uranium alphas should produce 167 000 and 147 000 electrons respectively, the energies being 4.763 MeV and 4.180 MeV. A 1.00 V test signal applied through C_T is calculated to induce 148 000 electrons on the collector,

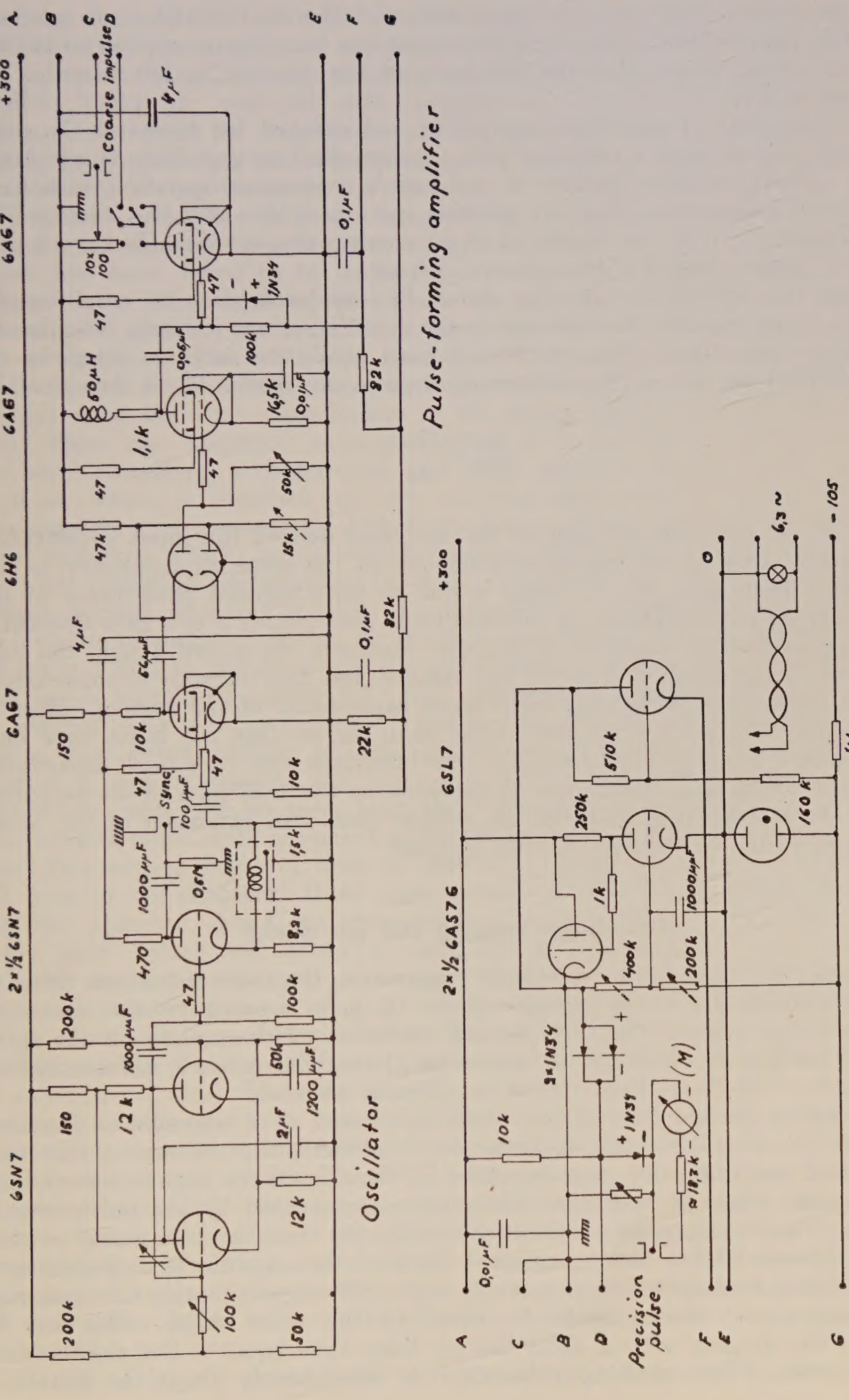


Fig. 6. Pulse generator for testing the stability of the apparatus.

using the value of C_T given on page 566. Relative to the alphas of uranium, using the figures above, this same test signal was found to correspond to 149 700 electrons, which shows, that the efficiency of the chamber is 100 % within the tolerance of C_T.

The constancy of the whole equipment was checked by means of the pulse generator, which gives a constant pulse, as described on page 564. It was found during several working periods of one week's continuous operation with fixed settings of all controls, that the greatest random drift about the average was approximately ± 1 ‰. In terms of alpha energy, this corresponds to ± 5 keV, which is good enough for the present purpose.

In this connection the following should be considered: since the amplifier plus chamber is principally an electrometer the most important property is constancy of effective capacitance C_{eff}, which is defined to be the collected charge on the input divided by the corresponding output pulse amplitude. C_{eff} is thus given by

$$C_{\text{eff}} = \frac{C_i}{A_0},$$

where A_0 is the voltage gain of the amplifier and C_i the input capacitance. It is clear, that C_i has the same influence on the resulting constancy as has the gain. That part of C_i which is due to the combined capacitance of the input tube is not stabilized by the feedback used (which is of a gain stabilizing sort) and cannot be expected to be very constant. As shown above, the constancy of C_{eff} is good in spite of this danger, but this certainly to some extent depends upon the rather big total input capacitance of the system. Most of this is due to rigid metal parts in the chamber, so that the input tube only contributes a minor part of the total. For chambers with very small capacitance one ought to employ other sorts of feedback in the first loop of the amplifier, tending to stabilize C_{eff} and not the veltage gain. An extension of the Miller integrator would be suitable for this purpose

III. Pulse forming and gas purity

Besides stability of the electronic apparatus, the most important thing in such measurements is the proper choice of pulse forming circuits since this influences that part of the total loss of resolution which originates in the apparatus. This loss of resolution is caused by 1) amplifier noise, 2) different collection times of electrons from tracks in different places.

Considering the amplifier noise, which is reduced to a minimum as described on page 562, it still remains to form the amplified pulses in such a way that the loss of resolution due to noise added by the amplifier is kept to a minimum. At the same time, no important information represented by the pulses should be lost. Theoretical considerations show (12, 13) that the best signal-to-noise ratio is obtained when the transmitted band of the amplifier is limited to such values that a transmitted step wave emerges with approximately the same rise- and decay-times, which should be equal to the pulse width. This may be obtained by clipping with a delay-line of delay time equal to half the rise time of the pulse. (The resulting pulse-width is consequently about the double of the delay time of the network). Assuming now that the desired pulse-form has been achieved by means of suitable networks, it remains to make sure that the collection times in the chamber are such that all the electrons from all possible tracks are collected with certainty in a time short compared to the chosen rise time of the amplifier, since the resulting pulse amplitude should be strictly proportional to the total charge, and should not depend in any way on the positions of the tracks in the chamber.

Now taking the known very big mobilities of electrons in very pure argon, or argon mixed with CO_2, together with the natural rise time of the amplifier, which has been found to be 0.45×3 μs, it should be possible, with moderate voltages upon the chamber, to obtain the required collecting time when the natural rise time of the amplifier is used. On the other hand, however, it is not advisable to use a clipping time equally short since the pulse amplitude in that case becomes very dependent on the rise time of the amplifier. This depends upon the stray capacitances of the tubes, and is not stabilized by feedback. Since the amplifier noise contributes a comparatively small amount to the total straggling, a compromise was made and the clipping time chosen to be 6 μs, which is obtained by two cascaded delay-lines of commercial type. This pulse-forming results in the 14 keV standard deviation of the assumed Gaussian noise spectrum which is mentioned on page 562. With such a clipping time the total collecting process need be short only compared to the clipping time.

Most workers prescribe the use of some chemical purification of the chamber gas (calcium or copper ovens etc. (3, 14)) to remove traces of oxygen and water from the argon. This is awkward and the purification is a very slow process. Since tank argon containing merely 1.7 p.p.m. of oxygen is available and rather cheap, we started with the procedure of flowing argon slowly through the chamber (via a short tube), without purification. The whole gas-system was high-vacuum tight so it seemed possible that after this process had been continued for some time there would be no more than the 1.7 p.p.m. of oxygen in the chamber. This worked fairly well; nevertheless, we observed detectable effects of impurities even after several weeks of work in this way, during which time no other gas had been in the chamber. If the argon stream through the chamber was stopped and the chamber closed, we observed that the resolution of the spectrometer slowly grew better and better, as compared to the stationary value obtained with the argon flowing, until a new and much better stationary condition was obtained after 8–10 hours "aging" of the chamber gas. The effect was such that a certain alpha-peak kept under observation became narrower and higher, the high energy flank remaining approximately in the same place. Fig. 7 shows an experiment which has been made in order to demonstrate this effect until stationary conditions have been obtained. It has not been possible due to practical circumstances to continue this experiment for a longer time, but more lengthy observations of the effect (one of three weeks duration) have been made in several cases with other samples, and, after the first 10 hours, all of these show no changes apart from statistical fluctuations. We think, that this is due to too big collecting time caused by the oxygen present in the chamber at the beginning of a run. It is possible, that the oxygen sticks to the highly polished and very clean inside walls of the chamber, being either adsorbed or chemically bound (20).

We did a lot of experiments to find out what happened in the chamber

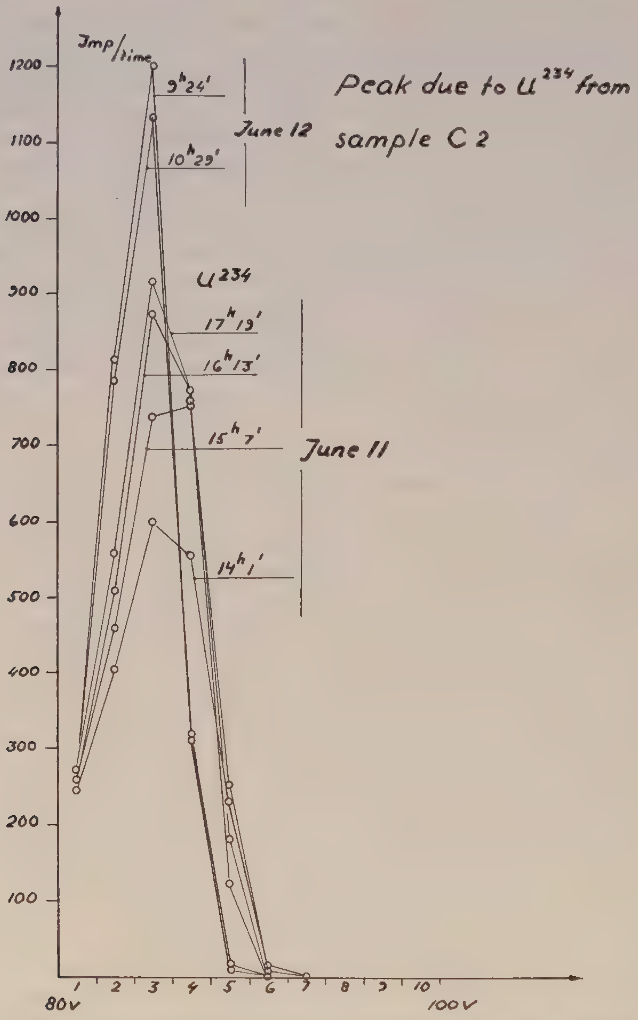


Fig. 7. Aging of argon with chamber closed. The chamber was closed at $13^h22'$, June 11., after argon had been flowing through it until stationary conditions were obtained. The points on each curve were measured during 1 hour the mean time of which is shown. The two curves of June 12th show the stationary condition after the aging.

during the "aging", but did not find any better explanation than that given above. During this work we observed that a number of organic vapours added to the tank argon in very small amounts (probably below 2 %) had exactly the same effect as the aging, apparently increasing the drift velocity considerably, even with the oxygen impurity present. If more vapour is added, but still only a few percent, the average work to produce an ion pair is decreased. Still bigger amounts completely stopped fast collection. This effect is presumably of the same kind as that caused by small amounts of CO_2, as described by ALLEN and ROSSI (15).

IV. Treatment of the data and results

Examples of the uranium spectra are shown in Figs. 8, 9. The question now is how to make the best possible energy determination from these curves. It is so, that no particular point on a certain alpha-peak can be said to represent the true energy, as there are a number of factors tending to "smear" it. These factors are of two kinds: 1) those influencing the collection of and subsequent measurement of the ionization products, 2) those influencing the energy of the emitted alpha particle. The first group comprises amplifier noise and all sorts of errors in the collection process such as induced charges, electron capture, or too long collecting time. The second group comprises the absorption in the sample causing an energy loss which is not measured. The first factors have the same influence on all pulses measured while the second ones have an influence changing with the thickness of the sample and the starting energy of the alpha-particle. The "smearing" caused by 1) and 2) are of approximately the same magnitude.

An analytical expression for a peak due to a certain alpha energy from a certain sample has to be computed by means of

$$P(E) = \int_0^E P_1(E-u) \cdot P_2'(u)\, d\, u,$$

where E is the energy and $P(E)$ the ordinate of the peak corresponding to the abscissa E. $P_1(E)$ and $P_2(E)$ are the peaks one would get with the same sample and energy of alpha particles if the factors 2) or 1) respectively were absent. Peaks of the same shape (being proportional) due to different alpha energies from a certain sample can be expected only when the absorption in the layer, represented by $P_2(E)$, is independent of the starting energy of the particles. Consequently in order to calculate the true energy corresponding to a certain peak by means of the analytical expression, this expression would have to be constructed separately for each peak even within the same spectrum, the information necessary for doing this being contained in the measured peak alone.

The only practical way in this case is to assume that the peaks belonging to a certain sample are nearly of the same shape. If this holds one has to find a characteristic point upon each peak and preferably one which is determined mainly by the high central part of the curve alone. As such may serve for instance the maximum or the center of gravity of the area. This point whose position relative to the true energy is fixed when the curves are of the same shape is assumed to represent the true energy.

In evaluating the spectra measured, both sorts of characteristic points have been used with very little difference in results; the maximum of the curves has been found by means of graphical differentiation. In the present case, the two big peaks are known to be due to U^{238} and U^{234}, whose energies are accurately known. The little one between them is due to U^{235} whose energy may be computed from the position of the maximum relative to the maximum points of the big peaks, since the energy scale of the spectra is strictly linear.

Since the whole range of energies under discussion is small (12 % of the maximum value) it may be hoped that the assumption of proportional peaks is

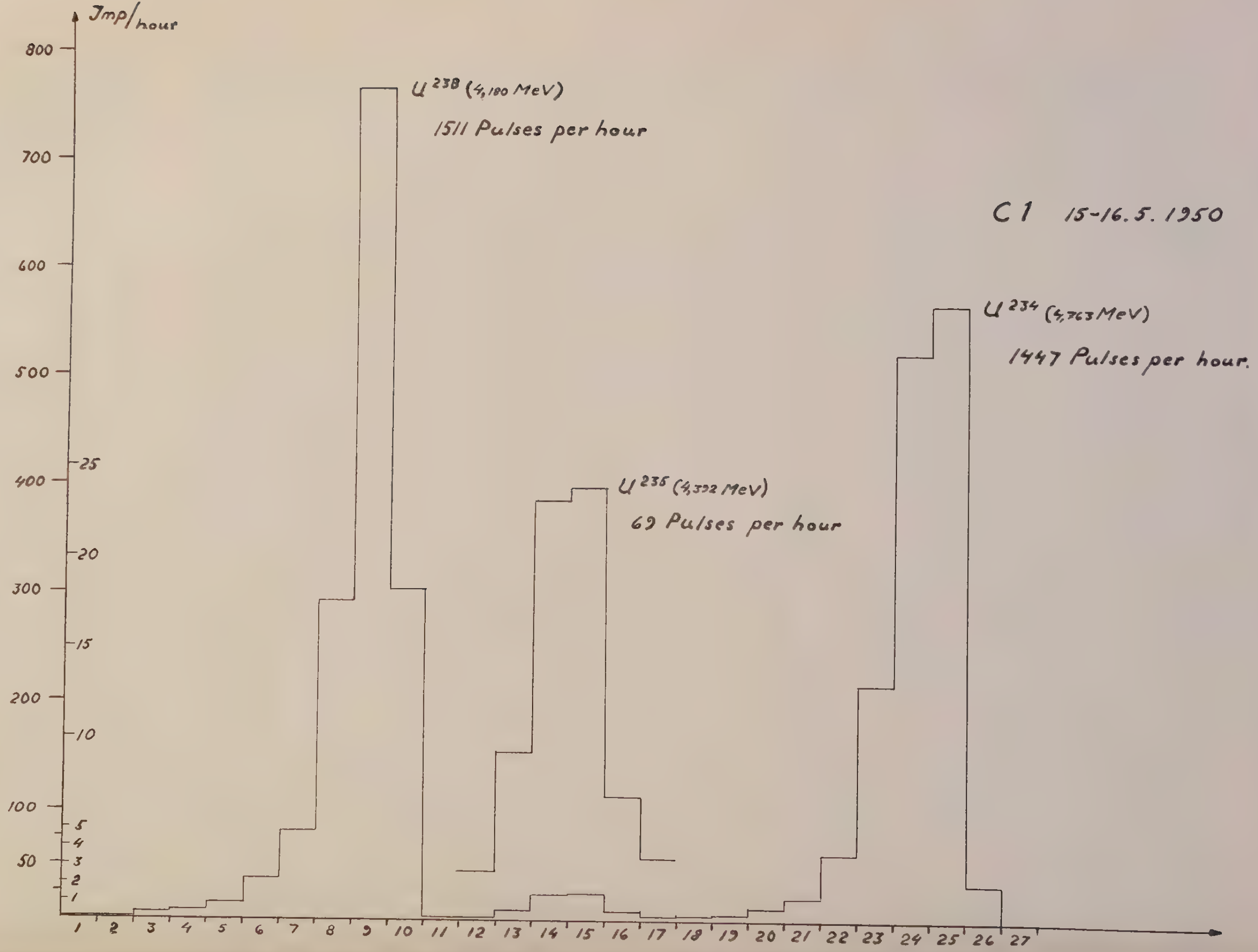


Fig. 8. Spectrum of sample C 1. The U^{235} peak is measured during 1 hour

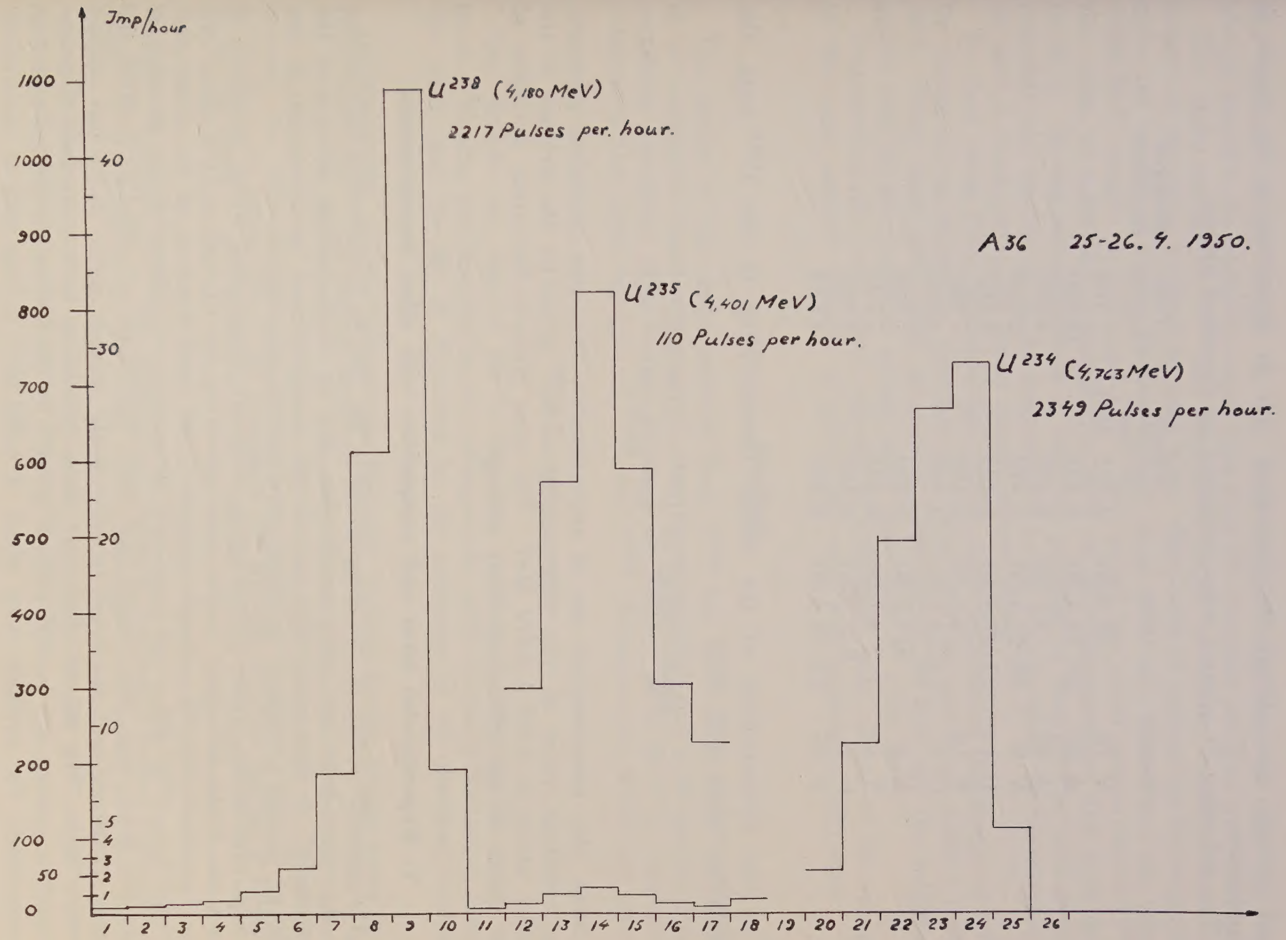


Fig. 9. Spectrum of sample A 36. The U^{235} peak is measured during 3 hours.

good enough even though the "smearing" due to absorption in the sample is appreciable.

The following 11 spectra were evaluated as described above:

Sample	Date	Energy of U^{235}	Total activity	Measured Imp./hour
V 10	24.4.50	4.371 MeV	7 700	”
A 38	23/24.4.50	4.421 ”	14 600	”
A 36	5.7.50	4.380 ”	4 395	”
V 8	26/27.4.50	4.372 ”	2 050	”
D 1	27/28.4.50	4.392 ”	5 157	”
V 10	2.5.50	4.400 ”	7 666	”
C 1	15/16.5.50	4.392 ”	3 039	”
U 1	5/8.5.50	4.400 ”	928	”
V 10	4.7.50	4.404 ”	7 723	”
C 2	6/7.5.50	4.392 ”	5 555	”
A 36	25/26.4.50	4.401 ”	4 694	”

In the determination of the energy corresponding to the U^{235} peak, the following values were used:

$$\begin{aligned} &\text{Alpha energy of } U^{238} = 4.180 \text{ MeV} \\ &\quad ” \quad\quad ” \quad\quad ” \; U^{234} = 4.763 \;\; ” \quad (17) \end{aligned}$$

Since the measurements are of approximately the same statistical goodness, the resulting value of the alpha energy of U^{235} given by the average of the values above is *4.393 ± 0.014 MeV*. The error given is the r.m.s. deviation of the values in the table from their average.

V. Experimental error and comparison with other measurements

The possible errors of the measurements are due to 1) statistical fluctuations in the pulse-rate and pulse-amplitudes, and to small random changes of the equipment, 2) systematic changes of the energy caused by absorption in the sample, pile-up of pulses, background in the chamber etc.

The systematic errors appearing as different shapes and wrong positions of the three peaks belonging to the same spectrum are of such a kind that they must increase when the sample is made more thick (because of the absorption in the layer) or when the pulse-rate is increased (pile-up). Since all samples have the same area, this means that there should be a correlation between the energy measured and the total activity of the sample given in the last column in the table. We cannot find any such correlation so we conclude that the deviations present are caused almost entirely by the factors mentioned under 1). Consequently the average value given of 4.393 MeV is true with an uncertainty which may be represented by the r.m.s. deviation from the average of the values measured.

The background of other pulses, which is not considered above, is so small that it is of no importance; moreover, it seems to be evenly distributed throughout the uranium energy range. It has been checked from time to time but was always found to be negligibly small (less than 0.5 impulses per hour and channel).

The alpha energy of U^{235} has been determined by others, in some cases by means of the same method as used here. The table of isotopes by Seaborg (16) gives the results of Clark, Spencer-Palmer et al. (17), who have found the value 4.396 MeV relative to U^{238} and U^{234}. They report two alpha groups due to U^{235}, the value of 4.396 MeV occurring in 80 % of all disintegrations and 4.56 MeV in 20 %. In our measurements it was not possible to discover any such group; probably the resolution of the spectrometer is too poor to allow the measurement of such a weak group by alpha measurements alone.

In their work (18), Baldinger and Huber have determined the energy of U^{235} by means of an ionization chamber and pulse analyzer and found 4.393 ± 0.015 MeV. They have also used the values 4.180 and 4.763 MeV for the reference points and do not mention values other than that of 4.393 MeV.

Disregarding the very weak activity of 4.56 MeV, both values of the energy of the main activity referred to above are very well consistent with ours.

Abstract

The energy of the alpha particles from U^{235} has been determined by means of an ionization chamber connected to a linear amplifier and a 10-channel pulse amplitude analyzer. The value obtained of 3.393 ± 0.014 MeV is measured relative to the alpha energies of U^{234} and U^{238}. Experimental details are given.

Acknowledgements

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